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**Title: Method and Apparatus for Manufacturing
High-Purity Aromatic Dicarboxylic Acid**

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(21) Application No.:	8-192495	(71) Applicant: 000165273
(22) Date of Filing:	July 22, 1996	Tsukishima Kikai Co., Ltd. 17-15 Tsukuda 2-chome, Chuo-ku, Tokyo
		(72) Inventor: Kenichi Hibi c/o Tsukishima Kikai Co., Ltd. 17-15 Tsukuda 2-chome, Chuo-ku, Tokyo
		(72) Inventor: Kazuo Okamoto c/o Tsukishima Kikai Co., Ltd. 17-15 Tsukuda 2-chome, Chuo-ku, Tokyo
		(72) Inventor: Keizo Takegami c/o Tsukishima Kikai Co., Ltd. 17-15 Tsukuda 2-chome, Chuo-ku, Tokyo
		(74) Agent: Yoshihisa Nagai, Patent Attorney (and 1 other)

(Continued on last page)

(54) [Title of the Invention]

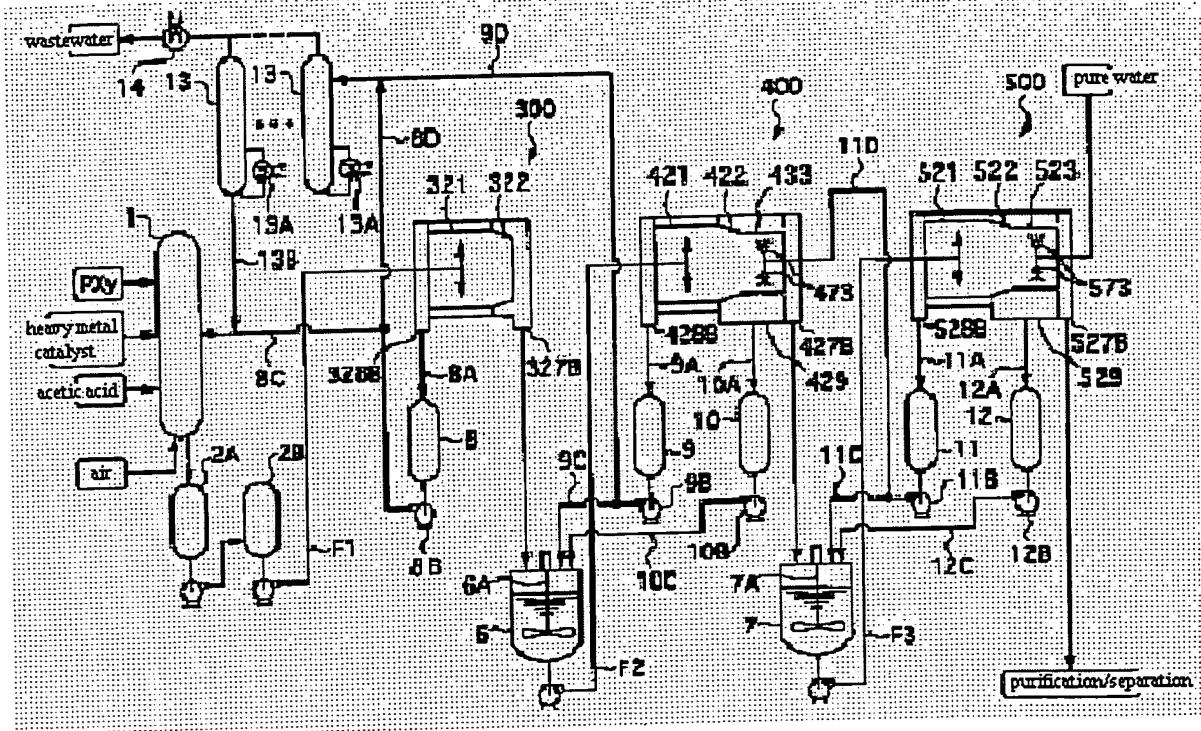
Method and Apparatus for Manufacturing
High-Purity Aromatic Dicarboxylic Acid

(57) [Summary]

[Object] To increase manufacturing efficiency by omitting a dryer and silo from a CTA recovery step, and to increase the purity of the resulting crystals.

[Means of Achievement] An acetic acid slurry of CTA from an oxidizing reaction 1 is fed to a solid-liquid separation/washing system in which a decanter centrifuge 300, a first screen/bowl decanter centrifuge 400, and a second screen/bowl decanter centrifuge 500 are disposed in the

sequence indicated, and a first reslurry tank 6 and a second reslurry tank 7 are disposed therebetween; the acetic acid is substituted by water, and the substituted cake is fed in unaltered form to the CTA purification/separation step of a subsequent stage.



[Claims]

[Claim 1] A method for manufacturing high-purity aromatic dicarboxylic acid having a crude aromatic dicarboxylic acid production/recovery step by an oxidation reaction in the presence of a reaction solvent and a catalyst in a preceding stage, and a purification/separation step for obtaining high-purity aromatic dicarboxylic acid from the crude aromatic dicarboxylic acid in a subsequent stage; said method for manufacturing an aromatic dicarboxylic acid characterized in that:

the crude aromatic dicarboxylic acid slurry in the reaction solvent produced by the oxidation reaction is fed to solid-liquid separation means and separated by the reaction solvent, and the separated cake is resuspended by first resuspension means;

the resuspended slurry from the first resuspension means is fed to a first screen/bowl decanter centrifuge, a solvent composed of the reaction solvent and water is separated in the first screen/bowl decanter centrifuge, washing and filtration are performed with the aid of a first washing solution, and the washed cake is resuspended by second resuspension means;

the resuspended slurry from the second resuspension means is fed to a second screen/bowl decanter centrifuge, an aqueous solvent whose main component is water is separated in the second screen/bowl decanter centrifuge, washing and filtration are performed with virtually clean water as a second washing solution, and the water-washed cake is fed in unaltered form to the purification/separation step of the subsequent stage;

the aqueous solvent separated in the second screen/bowl decanter centrifuge is used as the first washing solution, and the aqueous solvent and a filtrate of the second washing solution are used as the medium for the resuspended slurry in the second resuspension means; and

a solvent composed of water and the reaction solvent separated in the first screen/bowl decanter centrifuge, as well as a filtrate of the first washing solution, are used as the medium for the resuspended slurry in the first resuspension means.

[Claim 2] The method according to claim 1, wherein the water-washed cake from the second screen/bowl decanter centrifuge contains 0.1% by weight or less of an oxidation reaction solvent in relation to the aromatic dicarboxylic acid in the cake.

[Claim 3] The method according to claim 1 or 2, wherein the aromatic dicarboxylic acid is terephthalic acid.

[Claim 4] An apparatus for manufacturing high-purity aromatic dicarboxylic acid having crude aromatic dicarboxylic acid production/recovery means by an oxidation reaction in the presence of a reaction solvent and a catalyst in a preceding stage, and purification/separation means for obtaining high-purity aromatic dicarboxylic acid from the crude aromatic dicarboxylic acid in a subsequent stage; said apparatus for manufacturing high-purity aromatic dicarboxylic acid characterized in having:

means for feeding the crude aromatic dicarboxylic acid slurry in the reaction solvent produced by the oxidation reaction to solid-liquid separation means;

means for separating the reaction solvent in the solid-liquid separation means;

means for resuspending the separated cake in first resuspension means;

means for feeding the resuspended slurry from the first resuspension means to a first screen/bowl decanter centrifuge;

means for separating a solvent composed of the reaction solvent and water in the first screen/bowl decanter centrifuge and performing washing and filtration with the aid of a first washing solution;

means for resuspending the washed cake by second resuspension means;
means for feeding the resuspended slurry from the second resuspension means to a second screen/bowl decanter centrifuge;
means for separating an aqueous solvent whose main component is water in the second screen/bowl decanter centrifuge and performing washing and filtration with virtually clean water as a second washing solution; and
means for feeding the water-washed cake in unaltered form to the purification/separation step of the subsequent stage; and further comprising:
means for using the aqueous solvent separated in the second screen/bowl decanter centrifuge as the first washing solution;
means for using the aqueous solvent and a filtrate of the second washing solution as the medium for the resuspended slurry in the second resuspension means; and
means for using a solvent composed of water and the reaction solvent separated in the first screen/bowl decanter centrifuge, as well as a filtrate of the first washing solution, as the medium for the resuspended slurry in the first resuspension means.

[Detailed Description of the Invention]

[0001]

[Technological Field of the Invention] The present invention relates to a method for manufacturing isophthalic acid, terephthalic acid, naphthalene dicarboxylic acid, and other such aromatic dicarboxylic acids, and particularly relates to a method and apparatus for manufacturing a high-purity aromatic dicarboxylic acid whereby the manufacturing steps can be simplified and the cost of equipment reduced.

[0002]

[Prior Art] In general terms, a method for manufacturing an aromatic dicarboxylic acid, particularly a method for manufacturing high-purity terephthalic acid used as a raw material for polyester fiber, polyester resin, or the like, or a raw material for PET bottles, involves performing manufacturing steps in two stages (isophthalic acid, often used as a plasticizer, is also manufactured by virtually the same process).

[0003] Specifically, in the production/recovery step in the preceding stage, p-xylene is blown with compressed air in the presence of acetic acid or another such lower fatty acid-based solvent and a heavy metal catalyst typified by Co, Mn, and Br, and the resulting product is subjected to an oxidation reaction at a high temperature and high pressure to obtain crude terephthalic acid (hereinafter also referred to as CTA); and in the purification/separation step in the subsequent stage, the crude terephthalic acid is purified by a hydrogenation-reduction treatment or the like, and high-purity terephthalic acid (hereinafter also referred to as PTA) is manufactured.

[0004] To summarize this process by using Fig. 12, crude terephthalic acid (CTA) is produced under the previously described conditions in an oxidation reactor 50. Next, the resulting product is cooled and crystallized in a crystallizer 51, then a slurry of a reaction solvent containing terephthalic acid crystals, the impurities produced during the oxidation reaction, and the catalyst is fed to a rotary drum filter or another such solid-liquid separator 52, the acetic acid is separated, and a terephthalic acid cake is washed with aqueous acetic acid or acetic acid to remove the accompanying impurities and catalyst. Then, the remaining acetic acid is evaporated and separated by a rotary dryer 53 equipped with heating tubes, and the resulting product is fed and stored in a temporary retention silo 54. This stored CTA is metered out and suspended in a suspender 55, and is then fed to the purification/separation step of the subsequent stage.

[0005] The slurry from the suspender 55 is fed to a hydrogenation column 56 at a high temperature and high pressure, part or all of the CTA is dissolved and reacted with hydrogen gas in the presence of a fixed catalyst, and the impurities accompanying the CTA (mostly 4-carboxy benzaldehyde, or 4CBA, as well as p-toluyllic acid and a tar-like colored substance) are removed. This purification involves converting the 4CBA into p-toluyllic acid or partially into benzoic acid, and decolorizing the colored substance. Then, the resulting product is cooled and crystallized in a crystallizer 57, washing and solid-liquid separation are performed with the aid of solid-liquid separators 58A and 58B, the impurities remaining in the mother liquor are ultimately removed, and the resulting product is dried in a heating tube-equipped rotary dryer 59 and fed to a silo 60 to be stored as PTA.

[0006]

[Problems to Be Solved by the Invention] However, with the conventional art, a large heating tube-equipped rotary dryer 53 and a temporary retention silo 54 must be installed between the

point at which the slurry containing impurities produced by the oxidation reactor 50 is purged of crystals by a solid-liquid separator and washed, and the point at which the resulting product is then fed to the purification/separation step of the subsequent stage, to completely separate the remaining acetic acid. As a result, the equipment costs greatly increase and the process is essentially divided into a preceding-stage step designed to produce and recover materials, and a subsequent-stage step designed to purify and separate the materials, making it impossible to perform the process as a series of steps and to yield high manufacturing efficiency.

[0007] The conventional examples shown below have been proposed as methods for recovering crystals that improve upon the drawbacks described above and make it possible to implement a continuous recovery step.

[0008] (1) Domestic Republication No. 6-502653 discloses a method for solid-liquid separation of a CTA slurry with the aid of a so-called BHS filter system.

[0009] (2) Japanese Patent Application Laid-Open No. 6-327915 discloses a method wherein a CTA slurry is fed to and filtered in a rotary filter having a cylindrical filter medium, then the wet cake on the filter medium is washed with a washing solution while the filter medium is rotated, the resulting filtrate is circulated, the wet cake is washed, and the process is repeated at appropriate intervals in the direction opposite the rotation of the filter medium.

[0010] However, in the conventional examples described above, the acetic acid cannot be completely removed, and pure CTA cannot be adequately fed to the purification/separation step of the subsequent stage. Therefore, it is difficult to regard the purity of the ultimately obtained crystals as sufficient.

[0011] In view of this, the main object of the present invention is to omit the dryer and silo from the CTA recovery step, to reduce equipment costs, to increase manufacturing efficiency by combining the former stage and the subsequent stage in a continuous step, and to increase the purity of the resulting crystals.

[0012]

[Means Used to Solve the Above-Mentioned Problems] In the present invention designed to achieve the above-mentioned objects, the invention according to claim 1 is a method for manufacturing high-purity aromatic dicarboxylic acid having a crude aromatic dicarboxylic acid production/recovery step by an oxidation reaction in the presence of a reaction solvent and a

catalyst in a preceding stage, and a purification/separation step for obtaining high-purity aromatic dicarboxylic acid from the crude aromatic dicarboxylic acid in a subsequent stage; the method for manufacturing high-purity aromatic dicarboxylic acid characterized in that the crude aromatic dicarboxylic acid slurry in the reaction solvent produced by the oxidation reaction is fed to solid-liquid separation means and separated by the reaction solvent, and the separated cake is resuspended by first resuspension means; the resuspended slurry from the first resuspension means is fed to a first screen/bowl decanter centrifuge, a solvent composed of the reaction solvent and water is separated in the first screen/bowl decanter centrifuge, washing and filtration are performed with the aid of a first washing solution, and the washed cake is resuspended by second resuspension means; the resuspended slurry from the second resuspension means is fed to a second screen/bowl decanter centrifuge, an aqueous solvent whose main component is water is separated in the second screen/bowl decanter centrifuge, washing and filtration are performed with virtually clean water as a second washing solution, and the water-washed cake is fed in unaltered form to the purification/separation step of the subsequent stage; the aqueous solvent separated in the second screen/bowl decanter centrifuge is used as the first washing solution, and the aqueous solvent and a filtrate of the second washing solution are used as the medium for the resuspended slurry in the second resuspension means; and a solvent composed of water and the reaction solvent separated in the first screen/bowl decanter centrifuge, as well as a filtrate of the first washing solution, are used as the medium for the resuspended slurry in the first resuspension means.

[0013] The invention according to claim 2 is the method according to claim 1 wherein the water-washed cake from the second screen/bowl decanter centrifuge contains 0.1% by weight or less of an oxidation reaction solvent in relation to the aromatic dicarboxylic acid in the cake.

[0014] The invention according to claim 3 is the method according to claim 1 or 2 wherein the aromatic dicarboxylic acid is terephthalic acid.

[0015] The method according to claim 4 is an apparatus for manufacturing high-purity aromatic dicarboxylic acid having crude aromatic dicarboxylic acid production/recovery means by an oxidation reaction in the presence of a reaction solvent and a catalyst in a preceding stage, and purification/separation means for obtaining high-purity aromatic dicarboxylic acid from the crude aromatic dicarboxylic acid in a subsequent stage; the apparatus for manufacturing high-purity aromatic dicarboxylic acid characterized in having means for feeding the crude aromatic

dicarboxylic acid slurry in the reaction solvent produced by the oxidation reaction to solid-liquid separation means; means for separating the reaction solvent in the solid-liquid separation means; means for resuspending the separated cake in first resuspension means; means for feeding the resuspended slurry from the first resuspension means to a first screen/bowl decanter centrifuge; means for separating a solvent composed of the reaction solvent and water in the first screen/bowl decanter centrifuge and performing washing and filtration with the aid of a first washing solution; means for resuspending the washed cake by second resuspension means; means for feeding the resuspended slurry from the second resuspension means to a second screen/bowl decanter centrifuge; means for separating an aqueous solvent whose main component is water in the second screen/bowl decanter centrifuge and performing washing and filtration with virtually clean water as a second washing solution; and means for feeding the water-washed cake in unaltered form to the purification/separation step of the subsequent stage; and further comprising means for using the aqueous solvent separated in the second screen/bowl decanter centrifuge as the first washing solution; means for using the aqueous solvent and a filtrate of the second washing solution as the medium for the resuspended slurry in the second resuspension means; and means for using a solvent composed of water and the reaction solvent separated in the first screen/bowl decanter centrifuge, as well as a filtrate of the first washing solution, as the medium for the resuspended slurry in the first resuspension means.

[0016]

[Working Examples] The method for recovering pure crystals according to the present invention will now be described in detail with reference to the accompanying diagrams.

[0017] Fig. 1 shows the CTA production/recovery step in which high-purity terephthalic acid is obtained, wherein p-xylene PXy as a raw material is fed to an oxidation reactor 1 along with, for example, acetic acid and a heavy metal catalyst. Air is blown into the oxidation reactor 1, whereby the p-xylene PXy is oxidized and CTA is produced.

[0018] This CTA partially separates into crystals during the reaction, while the residue is sequentially fed to evaporation crystallizers 2A and 2B as a slurry dispersed in acetic acid, where it is cooled to precipitate most of the terephthalic acid.

[0019] The acetic acid slurry of CTA from the crystallizer 2B is fed to a solid-liquid separation/washing system that includes a decanter centrifuge 300 as the solid-liquid separation

means referred to in the present invention, a first screen/bowl decanter centrifuge 400, a second screen/bowl decanter centrifuge 500, and a first reslurry tank 6 and second reslurry tank 7 disposed therebetween, the acetic acid is substituted with water, and the crystal purity is increased while the crystals are washed.

[0020] Referring to Fig. 3, the basic structure of the decanter centrifuge 300 includes a bowl 320 composed of a frustoconical part 322 whose wider base is connected to a cylindrical main body 321, a screw conveyor 330 having a spiral screw blade 332 of uniform pitch in the outer surface of a conveyor main body 331 in the bowl 320, and rotating means that is disposed in a casing 301 and that causes the bowl 320 and the screw conveyor 330 to rotate in the same direction but at different speeds.

[0021] With this rotation means, support axles 326A and 326B for the bowl ends and support axles 333A and 333B for the corresponding screw conveyor ends are coaxially supported by bearings 302A and 302B, rotational drive force is transmitted from a drive motor (not shown) to a pulley gear 303 to rotate the bowl 320, the rotational force is reduced with a reducing mechanism 304, and the screw conveyor 330 is rotated at a different speed than the bowl 320.

[0022] An acetic acid slurry S1 of CTA from the crystallizer 2B is fed to the decanter centrifuge 300 via a supply channel F1, is guided to a compartment 334 in the conveyor main body 331 via a feed pipe 305 in the decanter centrifuge 300, is accelerated to near the rotating speed of the bowl 320 by an acceleration chamber 335, and is passed via a through-hole (not shown) in the wall to be supplied between the cylindrical main body 321¹ and the screw conveyor 330.

[0023] Due to a relationship in which the acetic acid slurry S1 is placed in the centrifugal force field in the bowl 320 and the screw conveyor 330, the solids contained in the slurry S1 are forced to the inner side of the bowl 320 and scraped out as a cake from the frustoconical part 322, as shown in the drawing, due to the rotation of the screw conveyor 330, and the cake to be dewatered is discharged from a cake discharge port 327A and a discharge chute 327B.

[0024] Meanwhile, clear liquid separated from the solids forms at one end of the bowl 320, and this liquid is discharged from a clear liquid discharge port 328A and a discharge chute 328B provided with a weir plate 325. The clear liquid from the discharge chute 328B has a high acetic acid concentration, is stored in a tank 8 via a conduit 8A, and is then returned to the oxidation

¹ Translator's note: The original reads "cylindrical main body 320," which is probably a typographical error.

reactor 1 by a pump 8B via a return channel 8C. Also, the clear liquid stored in the tank 8 is fed as necessary through a return channel 8D to a return channel 9D, to be described later, is guided to a group of dewatering and distillation columns 13, 13···, and is then separated into water vapor and acetic acid in the group of dewatering and distillation columns 13, 13···. The acetic acid is returned to the oxidation reactor 1 via return channels 13B and 8C. The water vapor is condensed into wastewater by a condenser 14. The symbol 13A denotes a heater.

[0025] The cake discharged from the cake discharge chute 327B is charged into the first reslurry tank 6, is reslurried by a stirring device 6A with the aid of the filtrate of the first washing solution and a portion of the clear liquid supplied from the below-described first screen/bowl decanter centrifuge 400 to the first reslurry tank 6, and is then fed to the first screen/bowl decanter centrifuge 400 via a supply channel F2.

[0026] The same basic structure can be used as both the first and second screen/bowl decanter centrifuges referred to in the present invention, and this structure has a cylindrical screen unit with a screen on the wall surface of the smaller base of the frustoconical shape, which expands in relation to the decanter centrifuge. Specifically, to describe the structure using Fig. 4, in which the same symbols are used as in the first screen/bowl decanter centrifuge in Fig. 1, the casing 401a² contains a bowl 420 with a cylindrical main body 421, a cylindrical screen unit 423 that has a screen 424 for filtering out solids on its wall and that is smaller than the cylindrical main body 421, and a frustoconical linking part 422 that links these two bodies; and a screw conveyor 430 having a spiraling screw blade 432 on the outer surface of a conveyor main body 431 in the bowl 420. The basic structure also includes rotation means for rotating the bowl 420 and the screw conveyor 430 at different speeds in the same direction.

[0027] In the rotation means, support axles 426A and 426B for the ends of the bowl 420, and support axles 433A and 433B for the ends of the corresponding screw conveyor 430 are coaxially supported on bearings 402A and 402B, rotational drive force is transmitted from a drive motor (not shown) to a pulley gear 403 to rotate the bowl 420, the rotational force is reduced with a reducing mechanism 404, and the screw conveyor 430 is rotated at a different speed than the bowl 420.

[0028] The slurry S supplied from the first reslurry tank 6 through the supply channel F2 is guided through a feed pipe 405 into a compartment 434 of the conveyor main body 431 in the

² Translator's note: Indicated as "casing 401" in the original, which appears to be a typographical error.

first screen/bowl decanter centrifuge 400, and is then passed via a through-hole in the wall to be supplied between the cylindrical main body 421 and the screw conveyor 430.

[0029] Due to a relationship in which the slurry S is placed in the centrifugal force field in the bowl 420 and the screw conveyor 430, the solids contained in the slurry S are forced to the inner side of the bowl 420, are scraped out as a cake from the linking part 422, as shown in the drawing, due to the rotation of the screw conveyor 430, and are delivered to the cylindrical screen unit 423. In the cylindrical screen unit 423, the washing solution W pressurized by the centrifugal force and fed into the conveyor main body 431 through an external tube 472 disposed concentrically with the feed pipe 405 and provided with a washing solution supply tube 471 at one end thereof is distributed over the cake from washing solution sprayers 473, 473... disposed between the screw blades 432, whereby the cake is washed and filtered (centrifugally dewatered) therein due to the presence of the screen 424, and the filtrate of the first washing solution is discharged as a screen filtrate into a screen filtrate chute 429 as illustrated. The dewatering cake transferred to the screen unit 423 is discharged from a cake discharge port 427A and a discharge chute 427B disposed at the other end, while the clear liquid separated from the solids is discharged from a clear liquid discharge port 428A and a discharge chute 428B provided with a weir plate 425 and formed at the first end of the bowl 420.

[0030] The filtrate of the first washing solution recovered from the screen filtrate chute 429 is stored in a first washing filtrate tank 10 via a guiding tube 10A, and is then guided through a return channel 10C by a pump 10B to the first reslurry tank 6 as a reslurry medium.

[0031] Also, the clear liquid discharged from the clear liquid discharge chute 428B is stored in a first recovery tank 9 via a guiding tube 9A, a part thereof is then guided by a pump 9B through a return channel 9C to the first reslurry tank 6 as a reslurry medium, the residue is guided through a return channel 9D to a group of dewatering and distillation columns 13, 13... composed of a plurality of dewatering and distillation columns, the residue is separated into water vapor and acetic acid in the group of dewatering and distillation columns 13, 13..., the acetic acid is then returned to the oxidation reactor 1 via return channels 13B and 8C, and the water vapor is condensed into wastewater by a condenser 14.

[0032] The cake discharged from the cake discharge chute 427B is charged into the second reslurry tank 7. Part of the filtrate of the second washing solution and the clear liquid from the second screen/bowl decanter centrifuge 500 are fed to the second reslurry tank 7, are reslurried

by a stirring device 7A, and are then fed to the second screen/bowl decanter centrifuge 500 via a supply channel F3.

[0033] In the second screen/bowl decanter centrifuge 500, solid-liquid separation and washing are performed in virtually the same manner as in the first screen/bowl decanter centrifuge 400, except that the second washing solution that is fed to washing solution sprayers 573, 573... is separately supplied pure water. Therefore, the operational configuration of this arrangement does not need to be described. The second washing solution may be the solution used as the washing solution in the purification/separation step.

[0034] The filtrate of the second washing solution recovered from the screen filtrate chute 529 is stored in a second washing filtrate tank 12 via a guiding tube 12A, is then guided by a pump 12B through a return channel 12C to the second reslurry tank 7, and is used as the reslurry medium.

[0035] The clear liquid recovered from a clear liquid discharge chute 528B is guided through a guiding tube 11A to a second recovery tank 11, part of the liquid is then guided by a pump 11B through a return channel 11D to a washing solution sprayer 473 as the first washing solution in the first screen/bowl decanter centrifuge 400, and the residue is fed to the second reslurry tank 7 via a return channel 11C. The rate at which the liquid is returned via these return channels can be appropriately controlled by providing a flow rate regulating valve or the like. When the second reslurry tank 7 is fed with a clear liquid from the second screen/bowl decanter centrifuge in addition to the supply of the filtrate of the second washing solution, it is known to result in extremely high washing efficiency.

[0036] The cake discharged from a cake discharge chute 527B of the second screen/bowl decanter centrifuge 500 is washed with pure water, the solvent is converted from an acetic acid base to a pure water base, and the material can therefore be supplied directly to the purification/separation step of the subsequent stage without passing through a dryer or silo. Therefore, equipment costs for a dryer and silo are reduced.

[0037] Also, the decanter centrifuge 300 and the first reslurry tank 6 in the above example can be omitted, and the acetic acid slurry from the crystallizer 2B can be fed directly to the first screen/bowl decanter centrifuge 400 via the supply channel F4, as shown in FIG. 5.

[0038] In the first screen/bowl decanter centrifuge 400 used in this embodiment, the filtrate of the first washing solution recovered from the screen filtrate chute 429 is stored in the first washing filtrate tank 10 via the guiding tube 10A, is then guided by the pump 10B through a

return channel 10D to the group of dewatering and distillation columns 13, 13..., and is separated into water vapor and acetic acid in the group of dewatering and distillation columns 13, 13... .

The acetic acid is returned to the oxidation reactor 1 via the return channels 13B and 8C.

[0039] Meanwhile, the clear liquid from the clear liquid discharge chute 428B is stored in a first recovery tank 9 via the guiding tube 9A, is then returned by the pump 9B through the return channel 9D to the oxidation reactor 1, and, if necessary, is returned through a return channel 9E to the return channel 10D for guiding the filtrate of the first washing solution to the group of dewatering and distillation columns 13, 13... .

[0040] However, when the filtrate of the first washing solution is returned to the oxidation reactor 1 via the group of dewatering and distillation columns 13, 13... in this manner, a regular screen/bowl decanter centrifuge has low crystal recovery efficiency because the crystals become admixed with the screen solution during screen filtration, and considerable leakage occurs. Therefore, the first washing solution must be fed to the supply side of the first screen/bowl decanter centrifuge, for example, to the supply channel F2, and the clear liquid from the clear liquid discharge chute 428B must be returned to the oxidation reaction system. In this case, the filtrate of the first washing solution contains a large amount of water, so the clear liquid is diluted with water, a greater load is imposed on the group of dewatering and distillation columns 13, 13... , and it becomes impossible to recover or discharge the water in a smooth manner.

[0041] These drawbacks are substantially overcome in the embodiment shown in Fig. 1, wherein the decanter centrifuge 300 and the first reslurry tank are used. Since the filtrate of the first washing solution from the first screen/bowl decanter centrifuge is returned to the first reslurry tank 6 and used as the reslurry medium, the leaked crystals are again fed to the first screen/bowl decanter centrifuge, and the crystal recovery efficiency increases. More specifically, in the present embodiment, the crystal recovery efficiency reaches about 99.95%, which is far superior to an extruding filter or other such type of continuous filtering device, which is about 70 to 90%. The clear liquid separated in the decanter centrifuge is mostly acetic acid and contains a small amount of water as a byproduct of the oxidation reaction. The liquid can be directly returned to the oxidation reactor 1 by the pump 8B via the return channel 8C as previously described, and can also be smoothly returned to the oxidation reactor 1 with a small amount of water when

directed to the group of dewatering and distillation columns 13, 13... Therefore, water can be recovered or discharged smoothly.

[0042] Instead of the above-mentioned decanter centrifuge, various continuous solid-liquid separators can be used as the solid-liquid separation means referred to in the present invention, which essentially should have a low rate of leakage of solids into the filtrate and should have satisfactory cake dewatering properties. For example, a BHS filter, a Young filter, an Oliver filter, or another such so-called rotary drum filter can be used.

[0043] Purification/Separation Step of the subsequent stage A CTA cake obtained by the crude aromatic dicarboxylic acid production/recovery step of the former stage as described above is fed to the purification/separation step wherein the crude aromatic dicarboxylic acid of the subsequent stage is subjected to a hydrogenation-reduction treatment to obtain high-purity aromatic dicarboxylic acid. Conventional methods, for example, a hydrogenation-reduction treatment, precision oxidation treatment, recrystallization treatment, or the like can be used in unaltered form as the purification/separation step of the subsequent stage. An example of hydrogenation-reduction treatment is described with reference to Fig. 2.

[0044] Specifically, a CTA cake obtained by converting a solvent from acetic acid to water is resuspended in the second reslurry tank 7³ and is then fed to a hydrogenation-reduction reactor 31. The impurities in the CTA are removed by a reduction treatment as a result of a hydrogenation reaction in the hydrogenation-reduction reactor 31. Specifically, 4CBA is converted to p-toluyllic acid or is partially converted to benzoic acid, and the tar-like colored substance is decolorized. Next, crystallization is performed by evaporation and cooling in an appropriate number (for example, five) of serially disposed crystallizers 32, the crystallized slurry is passed in sequence through a third rotary drum filter 33 that operates at increased pressure and a fourth drum filter 34 that operates at normal pressure, and the solid and liquid fractions are separated. A centrifuge may be used instead of the third rotary drum filter 33. In the solid-liquid separation means of the subsequent stage, the solid and liquid fractions are removed, the product is washed, the impurities are finally removed, and the material is dried in a dryer 35, kept in a silo 36, and stored as a finished product.

³ Translator's note: The original reads "second reslurry tank 30," which is probably a typographical error.

[0045] Preferred Screen/bowl Decanter Centrifuge for the Present Invention On the other hand, the screen/bowl decanter centrifuge in the conventional example described above uses a washing solution sprayer and does not necessarily have high washing efficiency.

[0046] The reason for this is described with reference to Fig. 11, wherein the same symbols are used as for the first screen/bowl decanter. The solids contained in the slurry supplied between the cylindrical main body 421 and the screw conveyor 430 via a through-hole in the wall of the compartment 434 are forced to the inner side of the bowl 420 in the centrifugal force field and are delivered in sequence to the cylindrical screen unit 423 by the screw blade 432, whereupon the solids are delivered as a cake C so as to be scraped together by the screw blade 432, and the surface level profile of the cake C slants at an incline towards the right side of the diagram as illustrated. After the cake C is delivered while the level profile is maintained, the inclination in the linking part 422 has an additional effect of further increasing the level incline, and the cake is scraped out and delivered to the screen unit 423 as the incline increases. Since a screen 424 is formed in the screen unit 423, filtration (centrifugal dewatering) is performed therein, and the screen filtrate is discharged as illustrated. Moreover, an uneven distribution of the cake C on the surface of the screen 424 creates an area on the forward delivery side in the section with the screw blades 432, 432 where there is no or virtually no cake C. When the washing solution has been spread out in this state (including misting or spraying), the washing solution primarily flows along the incline of the surface of the cake C and cuts through the section of the screen 424 corresponding to the region with no cake C layer. The washing solution thus fails to pass through the cake C layer, and washing efficiency cannot be increased.

[0047] In view of this, the following embodiments (1) through (3), wherein means for making the cake C level uniform are provided to a conventional screen/bowl decanter centrifuge, are proposed for a screen/bowl decanter centrifuge with a high washing efficiency that is suitable for the manufacture of high-purity aromatic dicarboxylic acid according to the present invention.

[0048] (1) First Embodiment: Leveling and smoothing means whereby the pitch P3 of the screw blade 432A in the section extending from the linking part 422 to the first portion of the screen unit 423, or the pitch P2 of the screw blade 432A in the screen unit 423, is smaller than the pitch P1 of the screw blade 432 at one end (clear liquid expulsion side).

[0049] Fig. 6 shows the first embodiment of a preferred screen/bowl decanter centrifuge according to the present invention. Specifically, the pitch P_2 of the screw blade 432A in the screen unit 423 is smaller than the pitch P_1 of the screw blade 432 next to the linking part 422.

[0050] In conventional practice, the aforementioned pitches are the same. Moreover, in the first embodiment, the pitch of the screw blade 432A in the screen unit 423 is relatively small. The degree by which the pitch is made smaller is set primarily under conditions in which the cake C layer is formed entirely within the section of the screw blades 432A, 432A in the screen unit 423, and is made as level as possible (for example, about 95 to 35%).

[0051] An option may also be considered in which it is better for the pitches of all the screw blades to be smaller, in which case an optimal screw blade pitch still exists in terms of the treated amount, solid-liquid separability, and other properties of the apparatus of a given size, so the screw blade 432 must be provided at this pitch to the cylindrical main body 421 and the linking part 422. By contrast, such factors are few in the case of the screen unit 423, and it is possible to vary the pitch of the screw blade 432A.

[0052] The first embodiment does not exclude aspects in which the pitches of the screw blades 432, 432 in the cylindrical main body 421 and the linking part 422 differ from each other, and the first embodiment is limited only in terms of the relationship between the pitch of the screw blade 432A in the screen unit 423 and the pitch of the screw blade 432 next to the linking part 422.

[0053] Also, as long as the pitch of the screw blade 432A in the screen unit 423 is smaller than the pitch of the screw blade 432 next to the linking part 422, it is possible to reduce the pitch in at least one section rather than decreasing the pitches between all the screw blades 432A, 432A, and 432A, 432A... in the screen unit 423. Furthermore, the pitch of the screw blades 432A in the screen unit 423 can be varied, and can, for example, be sequentially reduced towards the other end (the side with the solid discharge port 427A).

[0054] Decreasing the pitch P_3 in the section (including the screen unit) extending from the linking part 422 to the first portion of the screen unit 423 (not shown) is also included in the first embodiment.

[0055] Thus, in the first embodiment as well as in the following second and third embodiments, the cake washing efficiency can be improved because the washing solution from the washing solution sprayers 473, ...473 invariably passes through the entire cake C without creating a section

in which the surface of the screen 424 is exposed and is devoid of a cake C layer between the screw blades 432, as in conventional practice.

[0056] (2) Second Embodiment: Leveling and smoothing means wherein a screen may or may not be provided between the screw blade 432A next to the linking part 422 in the screen unit 423 and the adjacent screw blade 432A, but the screen 424 is blinded and a washing solution sprayer is provided facing the cake C surface.

[0057] Fig. 7 shows the second embodiment, wherein a blind member B is disposed between the screw blade 432A next to the linking part 422 in the screen unit 423 and the adjacent screw blade 432A.

[0058] In this case, the section impermeable to the washing solution in the screen unit 423 can have a pitch of 1/5 to 2 times the pitch P2 of the screw blade 432A, or of about 20 to 600 mm. In the example in Fig. 7, an area about 1.5 times the pitch serves as the section impermeable to the washing solution.

[0059] In the blind arrangement, it is possible to cover the screen 423 with a separate member while the screen is being formed, rather than directly extend the structural members of the linking part 422 by the corresponding length of the screen unit 423.

[0060] In the second embodiment, the region with the blind member B to which the washing solution is fed is not subjected to dewatering. Therefore, the amount of fluid in the cake C increases in this portion, and the cake C expands into the entire space between the screw blades 432A, 432A as a result of the increased fluidity. The smoothed level profile of the cake C is maintained even as the cake moves to the other end (the side with the solid discharge port 427A), and the cake washing efficiency is therefore improved in comparison with conventional practice.

[0061] (3) Third Embodiment: Leveling and smoothing means wherein the solid surface level between the screw blade 432A and the adjacent screw blade 432A is controlled and smoothing members M are provided to the screw conveyor 430.

[0062] Figs. 8 and 9 show the third embodiment. Specifically, the solid surface level between the screw blade 432A and the adjacent screw blade 432A is kept smooth, and smoothing members M are provided to the screw conveyor 430.

[0063] The smoothing members M may be fixed to the conveyor main body 431, or they may be fixed to the screw blade 432A. In either case, the distal edges of the smoothing members M in

the radial direction are fixed to leave a specific gap with the inner surface of the screen unit 423. Instead of being parallel with the axial direction of the apparatus, as shown in Fig. 8, the distal edges of the smoothing members M in the radial direction may form a straight or curved line at an appropriate angle. The smoothing members M may extend in the axial direction the entire length of the section with the screw blades 432A, 432A, or only part of the length, as shown in Fig. 8.

[0064] According to the third embodiment, the smoothing members M rotate along with the rotation of the screw conveyor 430, and the cake C on the screen unit 423 is smoothed into an entirely uniform thickness between the screw blades 432A, 432A.

[0065] (Other) In the second embodiment, it is essential that the washing solution be spread out, whereas in the other embodiments spreading out of the washing solution is desirable but not essential. Specifically, this is because smoothing the level of the cake C has the effect of facilitating centrifugal dewatering compared with when the level is not smoothed (the thickness of the layer when not smoothed offers resistance and reduces the centrifugal dewatering potential). In either case, in the method and apparatus for manufacturing high-purity terephthalic acid according to the present invention, the cake must be washed by the washing solution, and the first through third embodiments described above can therefore be suitably used.

[0066] A working example of the method for manufacturing high-purity terephthalic acid according to the present invention is shown below, and the effects of the present invention are described.

[0067] <Working Example> Fig. 10 shows an operating example of the CTA production/separation step according to the present invention. The weight ratio of the acetic acid, indicated as "A.A." in the diagram, in the slurry supplied from the oxidation reaction system to the decanter 300 can be such that the ratio, which is initially 58.60%, is then changed to A.A. = 14.82% for the slurry in the reslurry tank 6, to A.A. = 1.38% for the slurry in the reslurry tank 7, and to A.A. = 0.09% for the cake discharged from the second screen/bowl decanter centrifuge 500. It should be noted that A.A. contains many impurities due to the reaction.

[0068] Therefore, it is possible to dispense with the need for the conventionally used step of drying the material in a dryer and storing it in a silo, and to continuously perform the CTA manufacturing/recovery step and the purification/separation step.

[0069] Also, the above descriptions were focused on the manufacture of terephthalic acid, but those skilled in the art should easily understand without the aid of any working examples that the present invention can also be used in the manufacture of isophthalic acid, naphthalene dicarboxylic acid, and other such aromatic dicarboxylic acids.

[0070]

[Effect of the Invention] As described above, the present invention is advantageous in making it possible to dispense with the need for the drying step with a CTA dryer and the storage step with a CTA silo that have been used in conventional practice, to reduce the necessary equipment costs, to increase manufacturing efficiency by combining the preceding and subsequent stages into a single continuous step, and to improve the purity of the resulting crystals.

[Brief Description of the Drawings]

[Figure 1] A flowsheet of the terephthalic acid production/recovery step according to the present invention

[Figure 2] A flowsheet of the purification/separation step of the subsequent stage

[Figure 3] A longitudinal cross-sectional view showing an outline of the decanter centrifuge

[Figure 4] A longitudinal cross-sectional view showing an outline of a conventional screen/bowl decanter centrifuge

[Figure 5] A flowsheet showing another embodiment of the terephthalic acid production/recovery method according to the present invention

[Figure 6] A schematic enlarged longitudinal sectional view showing a first embodiment of a screen/bowl decanter centrifuge suitable for the present invention

[Figure 7] A schematic enlarged longitudinal sectional view showing a second embodiment of a screen/bowl decanter centrifuge suitable for the present invention

[Figure 8] A schematic enlarged longitudinal sectional view showing a third embodiment of a screen/bowl decanter centrifuge suitable for the present invention

[Figure 9] A cross-sectional side view thereof

[Figure 10] An explanatory diagram of an operating example

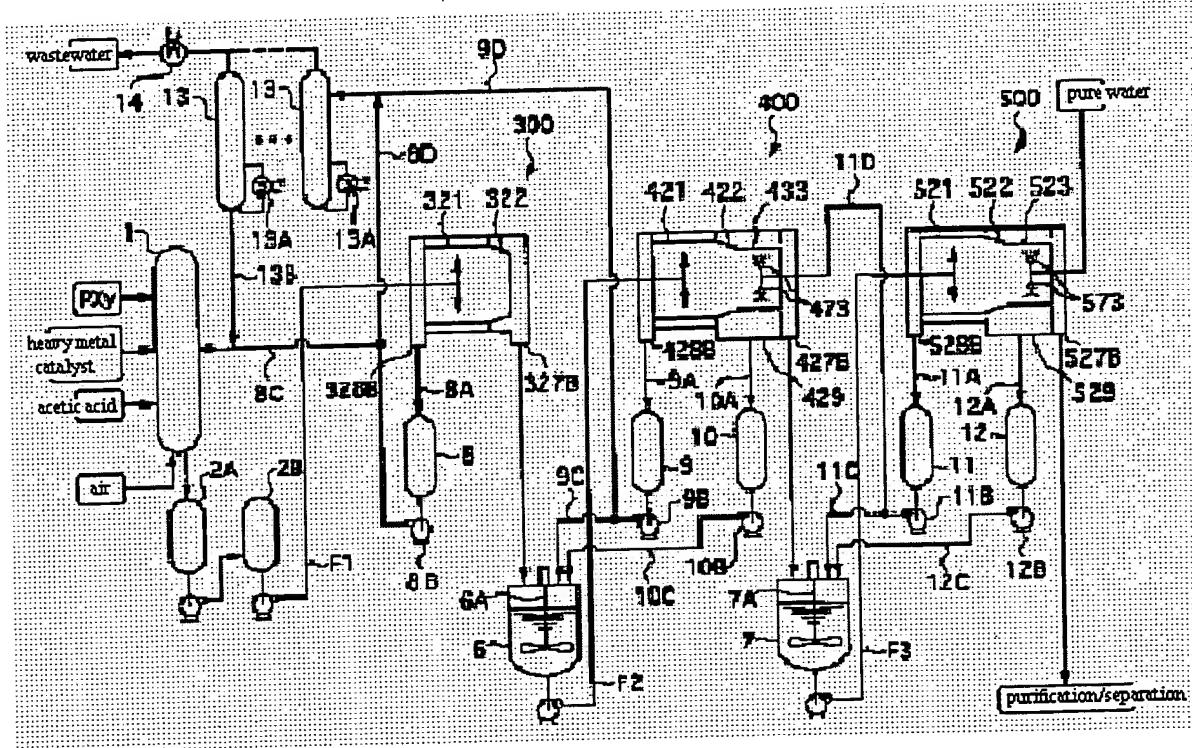
[Figure 11] A schematic enlarged longitudinal sectional view of Fig. 4

[Figure 12] A schematic flowsheet of a conventional example

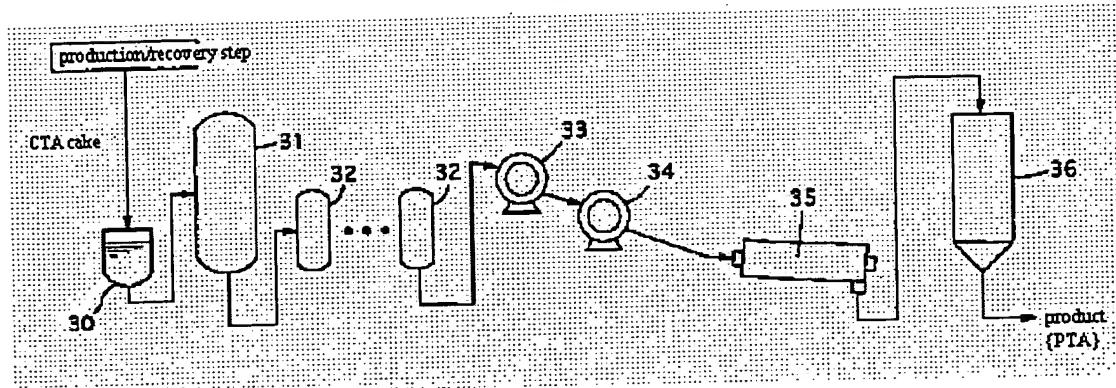
[Key]

1: oxidation reactor, 2A, 2B: evaporation crystallizer, 6: first reslurry tank, 7: second reslurry tank, 9: first recovery tank, 10: first washing filtrate tank, 11: second recovery tank, 12: second washing filtrate tank, 13: dewatering and distillation columns, 300: decanter centrifuge, 400: first screen/bowl decanter centrifuge, 500: second screen/bowl decanter centrifuge

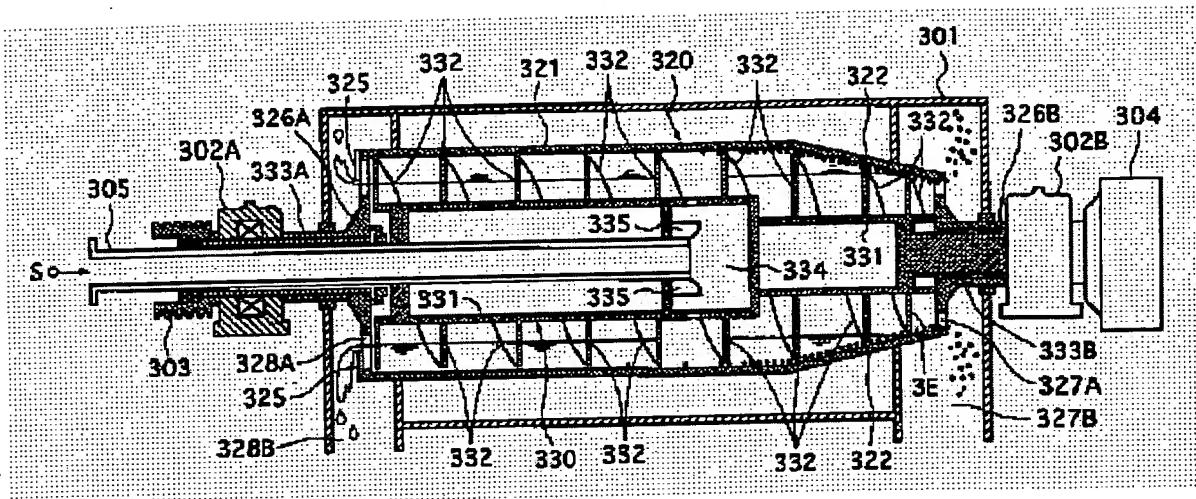
[Fig. 1]



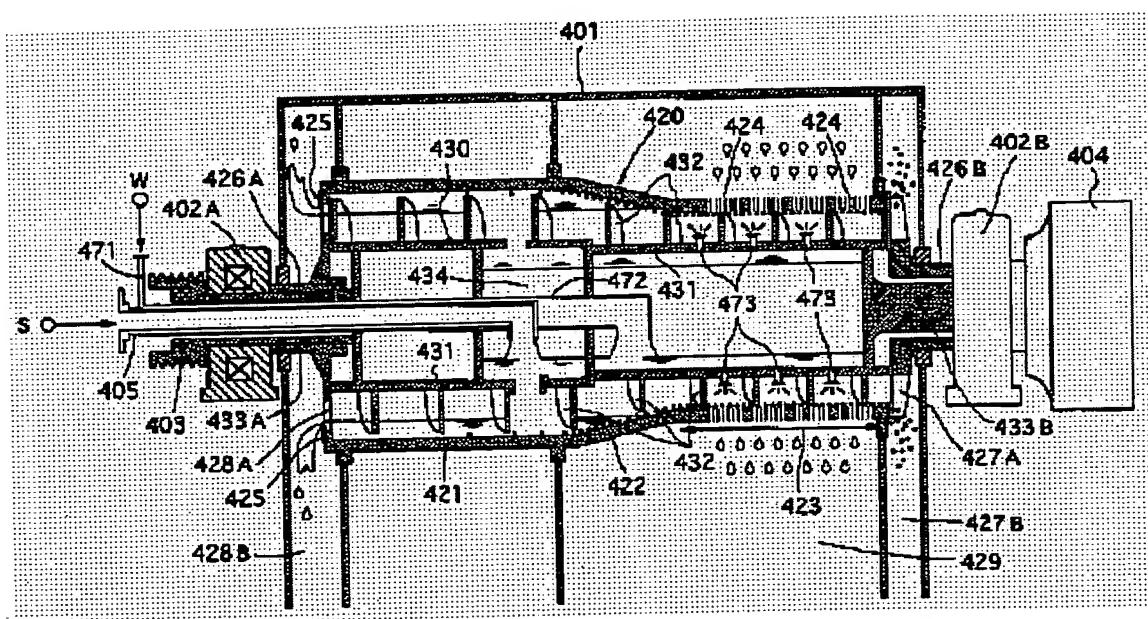
[Fig. 2]



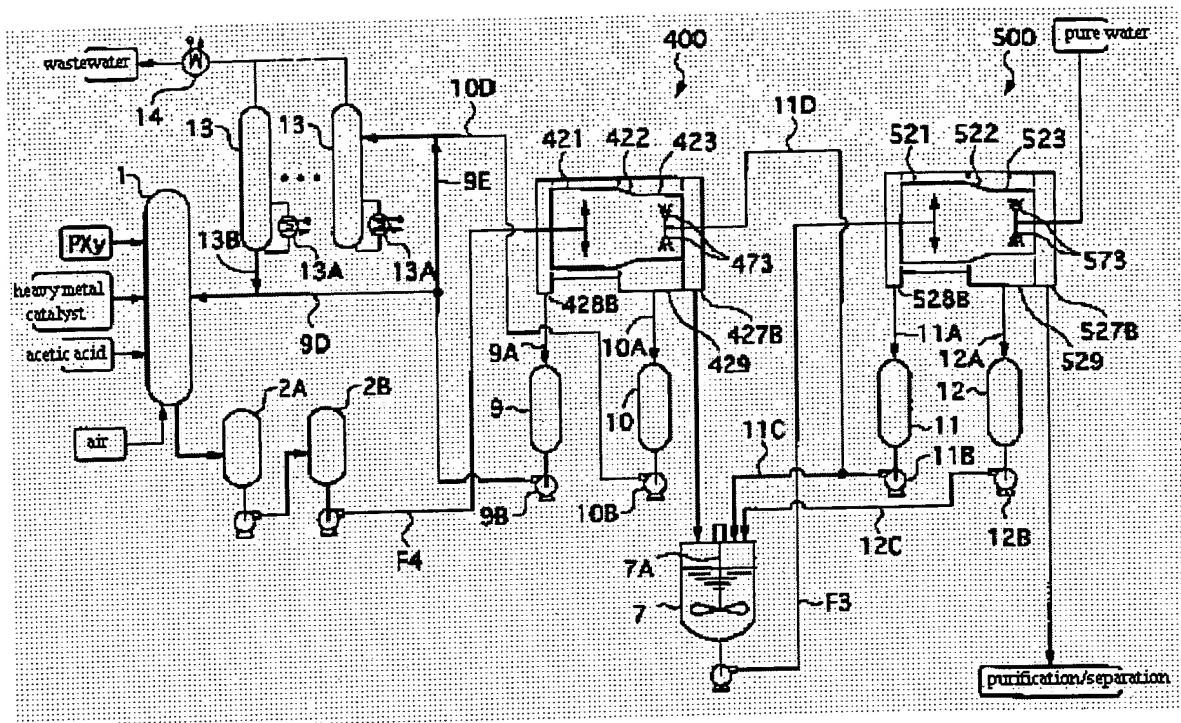
[Fig. 3]



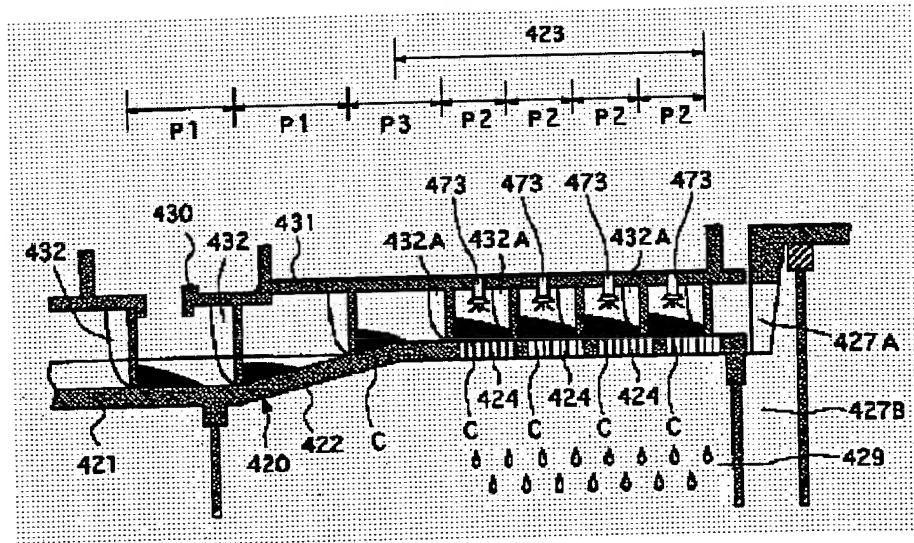
[Fig. 4]



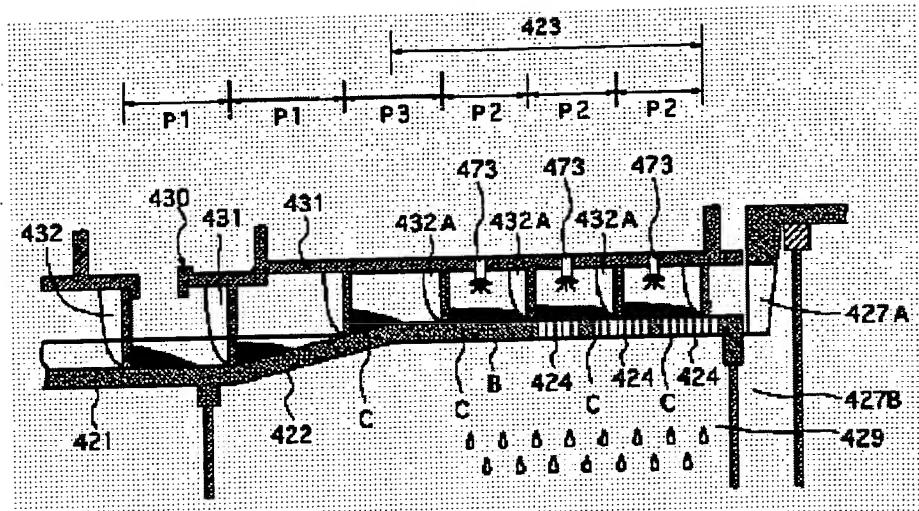
[Fig. 5]



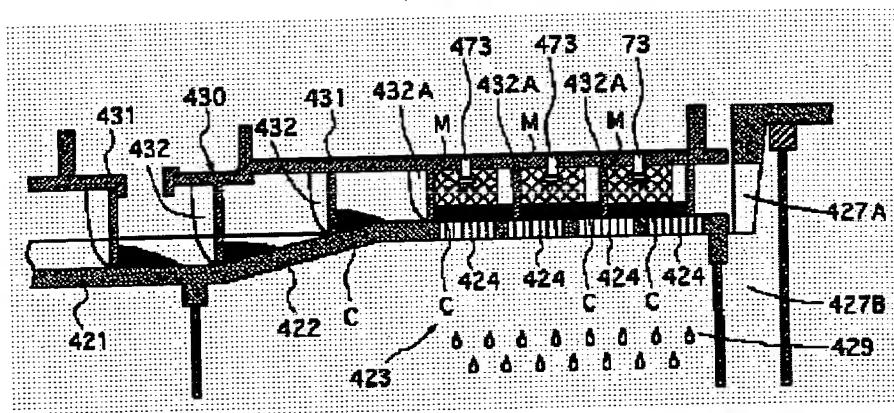
[Fig. 6]



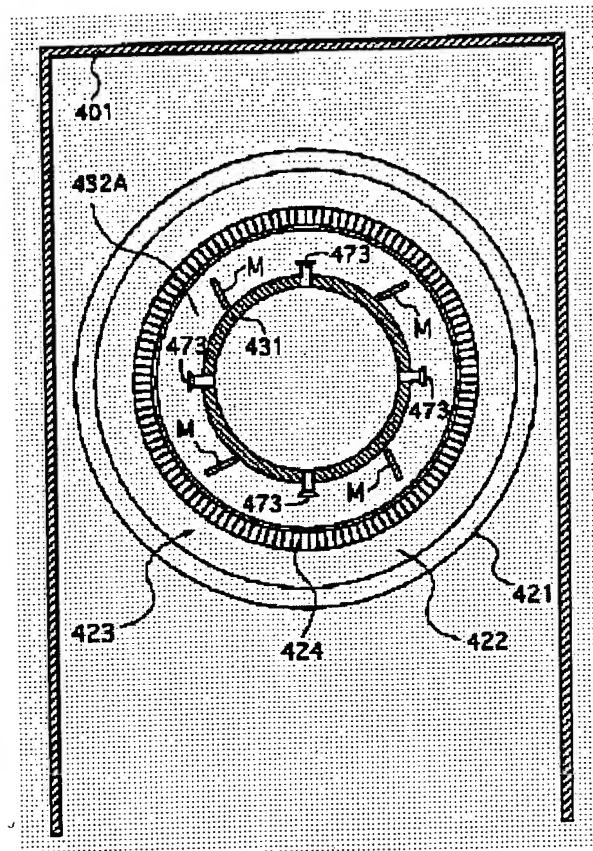
[Fig. 7]



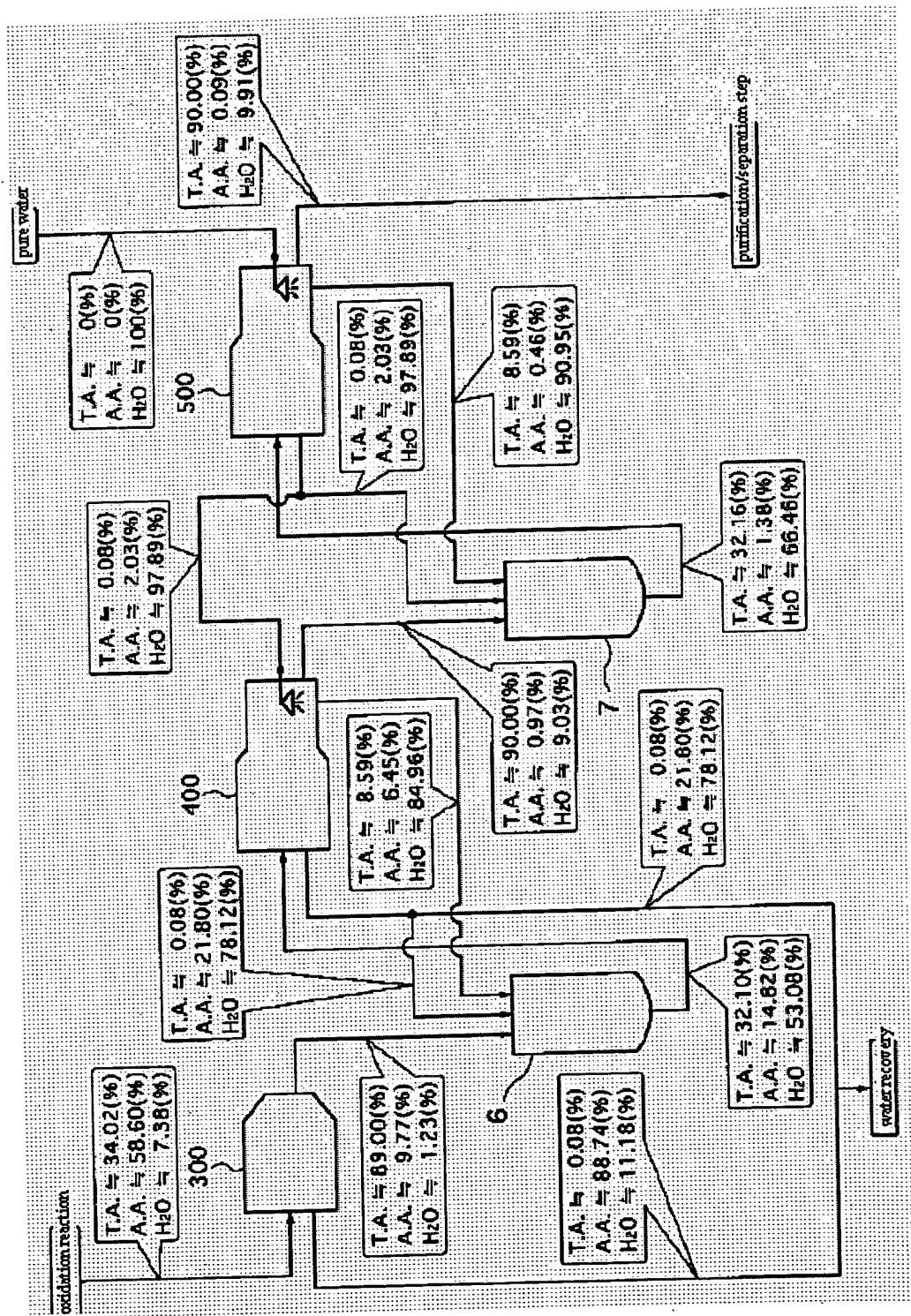
[Fig. 8]



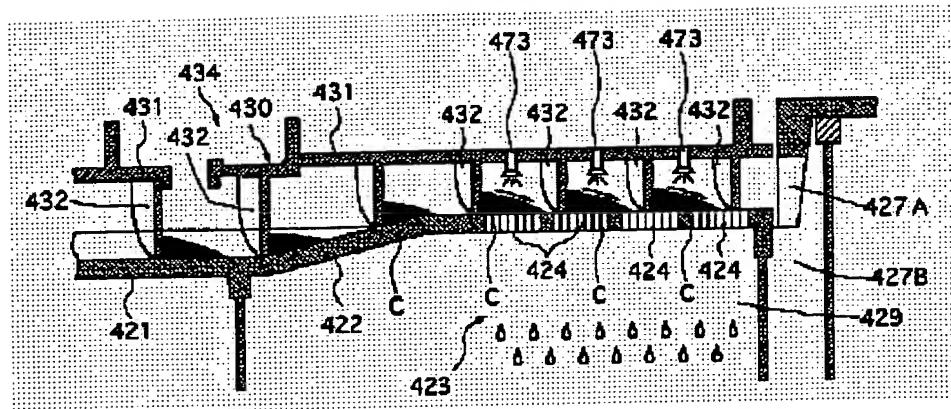
[Fig. 9]



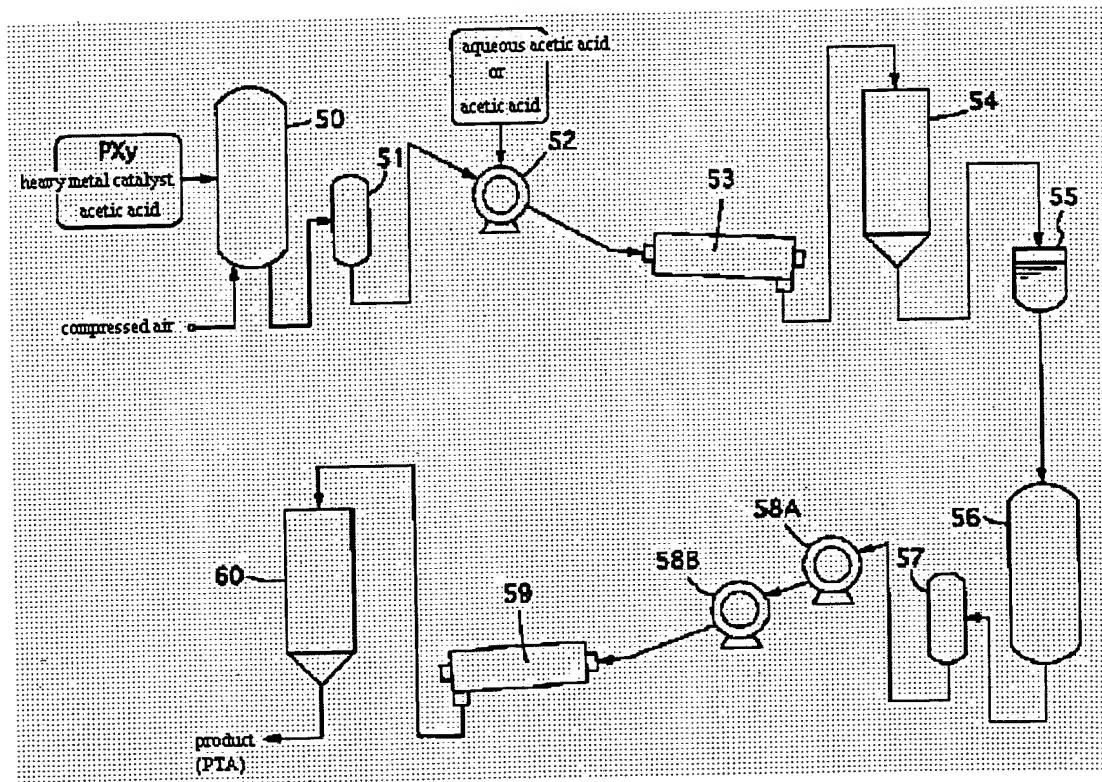
[Fig. 10]



[Fig. 11]



[Fig. 12]



(Continued from front page)

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(72) Inventor: Keizo Takegami
c/o Tsukishima Kikai Co., Ltd.
17-15 Tsukuda 2-chome, Chuo-ku, Tokyo

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[Document Amended] Specification

[Section Amended] Detailed Description of the Invention

[Amendment Method] Change

[Details of Amendment]

[Detailed Description of the Invention]

[0001]

[Technological Field of the Invention] The present invention relates to a method for manufacturing isophthalic acid, terephthalic acid, naphthalene dicarboxylic acid, and other such aromatic dicarboxylic acids, and particularly relates to a method and apparatus for manufacturing a high-purity aromatic dicarboxylic acid whereby the manufacturing steps can be simplified and the cost of equipment reduced.

[0002]

[Prior Art] In general terms, a method for manufacturing an aromatic dicarboxylic acid, particularly a method for manufacturing high-purity terephthalic acid used as a raw material for polyester fiber, polyester resin, or the like, or a raw material for PET bottles, involves performing manufacturing steps in two stages (isophthalic acid, often used as an plasticizer, is also manufactured by virtually the same process).

[0003] Specifically, in the production/recovery step in the preceding stage, p-xylene is blown with compressed air in the presence of acetic acid or another such lower fatty acid-based solvent and a heavy metal catalyst typified by Co, Mn, and Br, and the resulting product is subjected to an oxidation reaction at a high temperature and high pressure to obtain crude terephthalic acid (hereinafter also referred to as CTA); and in the purification/separation step in the subsequent stage, the crude terephthalic acid is purified by a hydrogenation-reduction treatment or the like, and high-purity terephthalic acid (hereinafter also referred to as PTA) is manufactured.

[0004] To summarize this process by using Fig. 12, crude terephthalic acid (CTA) is produced under the previously described conditions in an oxidation reactor 50. Next, the resulting product is cooled and crystallized in a crystallizer 51, then a slurry of a reaction solvent containing terephthalic acid crystals, the impurities produced during the oxidation reaction, and the catalyst is fed to a rotary drum filter or another such solid-liquid separator 52, the acetic acid is separated, and a terephthalic acid cake is washed with aqueous acetic acid or acetic acid to remove the accompanying impurities and catalyst. Then, the remaining acetic acid is evaporated and separated by a rotary dryer 53 equipped with heating tubes, and the resulting product is fed and

stored in a temporary retention silo 54. This stored CTA is metered out and suspended in a suspender 55, and is then fed to the purification/separation step of the subsequent stage.

[0005] The slurry from the suspender 55 is fed to a hydrogenation column 56 at a high temperature and high pressure, part or all of the CTA is dissolved and reacted with hydrogen gas in the presence of a fixed catalyst, and the impurities accompanying the CTA (mostly 4-carboxy benzaldehyde, or 4CBA, as well as p-toluyllic acid and a tar-like colored substance) are removed. This purification involves converting the 4CBA into p-toluyllic acid or partially into benzoic acid, and decolorizing the colored substance. Then, the resulting product is cooled and crystallized in a crystallizer 57, washing and solid-liquid separation are performed with the aid of solid-liquid separators 58A and 58B, the impurities remaining in the mother liquor are ultimately removed, and the resulting product is dried in a heating tube-equipped rotary dryer 59 and fed to a silo 60 to be stored as PTA.

[0006]

[Problems to Be Solved by the Invention] However, with the conventional art, a large heating tube-equipped rotary dryer 53 and a temporary retention silo 54 must be installed between the point at which the slurry containing impurities produced by the oxidation reactor 50 is purged of crystals by a solid-liquid separator 52 and washed, and the point at which the resulting product is then fed to the purification/separation step of the subsequent stage, to completely separate the remaining acetic acid. As a result, the equipment costs greatly increase and the process is essentially divided into a preceding-stage step designed to produce and recover materials, and a subsequent-stage step designed to purify and separate the materials, making it impossible to perform the process as a series of steps and to yield high manufacturing efficiency.

[0007] The conventional examples shown below have been proposed as methods for recovering crystals that improve upon the drawbacks described above and make it possible to implement a continuous recovery step.

[0008] (1) Domestic Republication No. 6-502653 discloses a method for solid-liquid separation of a CTA slurry with the aid of a so-called BHS filter system.

[0009] (2) Japanese Patent Application Laid-Open No. 6-327915 discloses a method wherein a CTA slurry is fed to and filtered in a rotary filter having a cylindrical filter medium, then the wet cake on the filter medium is washed with a washing solution while the filter medium is rotated,

the resulting filtrate is circulated, the wet cake is washed, and the process is repeated at appropriate intervals in the direction opposite the rotation of the filter medium.

[0010] However, in the conventional examples described above, the acetic acid cannot be completely removed, and pure CTA cannot be adequately fed to the purification/separation step of the subsequent stage. Therefore, it is difficult to regard the purity of the ultimately obtained crystals as sufficient.

[0011] In view of this, the main object of the present invention is to omit the dryer and silo from the CTA recovery step, to reduce equipment costs, to increase manufacturing efficiency by combining the former stage and the subsequent stage in a continuous step, and to increase the purity of the resulting crystals.

[0012]

[Means Used to Solve the Above-Mentioned Problems] In the present invention designed to achieve the above-mentioned objects, the invention according to claim 1 is a method for manufacturing high-purity aromatic dicarboxylic acid having a crude aromatic dicarboxylic acid production/recovery step by an oxidation reaction in the presence of a reaction solvent and a catalyst in a preceding stage, and a purification/separation step for obtaining high-purity aromatic dicarboxylic acid from the crude aromatic dicarboxylic acid in a subsequent stage; the method for manufacturing an aromatic dicarboxylic acid characterized in that the crude aromatic dicarboxylic acid slurry in the reaction solvent produced by the oxidation reaction is fed to solid-liquid separation means and separated by the reaction solvent, and the separated cake is resuspended by first resuspension means; the resuspended slurry from the first resuspension means is fed to a first screen/bowl decanter centrifuge, a solvent composed of the reaction solvent and water is separated in the first screen/bowl decanter centrifuge, washing and filtration are performed with the aid of a first washing solution, and the washed cake is resuspended by second resuspension means; the resuspended slurry from the second resuspension means is fed to a second screen/bowl decanter centrifuge, an aqueous solvent whose main component is water is separated in the second screen/bowl decanter centrifuge, washing and filtration are performed with virtually clean water as a second washing solution, and the water-washed cake is fed in unaltered form to the purification/separation step of the subsequent stage; the aqueous solvent separated in the second screen/bowl decanter centrifuge is used as the first washing solution, and

the aqueous solvent and a filtrate of the second washing solution are used as the medium for the resuspended slurry in the second resuspension means; and a solvent composed of water and the reaction solvent separated in the first screen/bowl decanter centrifuge, as well as a filtrate of the first washing solution, are used as the medium for the resuspended slurry in the first resuspension means.

[0013] The invention according to claim 2 is the method according to claim 1 wherein the water-washed cake from the second screen/bowl decanter centrifuge contains 0.1% by weight or less of an oxidation reaction solvent in relation to the aromatic dicarboxylic acid in the cake.

[0014] The invention according to claim 3 is the method according to claim 1 or 2 wherein the aromatic dicarboxylic acid is terephthalic acid.

[0015] The method according to claim 4 is an apparatus for manufacturing high-purity aromatic dicarboxylic acid having crude aromatic dicarboxylic acid production/recovery means by an oxidation reaction in the presence of a reaction solvent and a catalyst in a preceding stage, and purification/separation means for obtaining high-purity aromatic dicarboxylic acid from the crude aromatic dicarboxylic acid in a subsequent stage; the apparatus for manufacturing high-purity aromatic dicarboxylic acid characterized in having means for feeding the crude aromatic dicarboxylic acid slurry in the reaction solvent produced by the oxidation reaction to solid-liquid separation means; means for separating the reaction solvent in the solid-liquid separation means; means for resuspending the separated cake in first resuspension means; means for feeding the resuspended slurry from the first resuspension means to a first screen/bowl decanter centrifuge; means for separating a solvent composed of the reaction solvent and water in the first screen/bowl decanter centrifuge and performing washing and filtration with the aid of a first washing solution; means for resuspending the washed cake by second resuspension means; means for feeding the resuspended slurry from the second resuspension means to a second screen/bowl decanter centrifuge; means for separating an aqueous solvent whose main component is water in the second screen/bowl decanter centrifuge and performing washing and filtration with virtually clean water as a second washing solution; and means for feeding the water-washed cake in unaltered form to the purification/separation step of the subsequent stage; and further comprising means for using the aqueous solvent separated in the second screen/bowl decanter centrifuge as the first washing solution; means for using the aqueous solvent and a filtrate of the second washing solution as the medium for the resuspended slurry in the second

resuspension means; and means for using a solvent composed of water and the reaction solvent separated in the first screen/bowl decanter centrifuge, as well as a filtrate of the first washing solution, as the medium for the resuspended slurry in the first resuspension means.

[0016]

[Working Examples] The method for recovering pure crystals according to the present invention will now be described in detail with reference to the accompanying diagrams.

[0017] Fig. 1 shows the CTA production/recovery step in which high-purity terephthalic acid is obtained, wherein p-xylene PXy as a raw material is fed to an oxidation reactor 1 along with, for example, acetic acid and a heavy metal catalyst. Air is blown into the oxidation reactor 1, whereby the p-xylene PXy is oxidized and CTA is produced.

[0018] This CTA partially separates into crystals during the reaction, while the residue is sequentially fed to evaporation crystallizers 2A and 2B as a slurry dispersed in acetic acid, where it is cooled to precipitate most of the terephthalic acid.

[0019] The acetic acid slurry of CTA from the crystallizer 2B is fed to a solid-liquid separation/washing system that includes a decanter centrifuge 300 as the solid-liquid separation means referred to in the present invention, a first screen/bowl decanter centrifuge 400, a second screen/bowl decanter centrifuge 500, and a first reslurry tank 6 and second reslurry tank 7 disposed therebetween, the acetic acid is substituted with water, and the crystal purity is increased while the crystals are washed.

[0020] Referring to Fig. 3, the basic structure of the decanter centrifuge 300 includes a bowl 320 composed of a frustoconical part 322 whose wider base is connected to a cylindrical main body 321, a screw conveyor 330 having a spiral screw blade 332 of uniform pitch in the outer surface of a conveyor main body 331 in the bowl 320, and rotating means that is disposed in a casing 301 and that causes the bowl 320 and the screw conveyor 330 to rotate in the same direction but at different speeds.

[0021] With this rotation means, support axles 326A and 326B for the bowl ends and support axles 333A and 333B for the corresponding screw conveyor ends are coaxially supported by bearings 302A and 302B, rotational drive force is transmitted from a drive motor (not shown) to a pulley gear 303 to rotate the bowl 320, the rotational force is reduced with a reducing mechanism 304, and the screw conveyor 330 is rotated at a different speed than the bowl 320.

[0022] An acetic acid slurry S of CTA from the crystallizer 2B is fed to the decanter centrifuge 300 via a supply channel F1, is guided to a compartment 334 in the conveyor main body 331 via a feed pipe 305 in the decanter centrifuge 300, is accelerated to near the rotating speed of the bowl 320 by an acceleration chamber 335, and is passed via a through-hole (not shown) in the wall to be supplied between the bowl 320 and the screw conveyor 330.

[0023] Due to a relationship in which the acetic acid slurry S is placed in the centrifugal force field in the bowl 320 and the screw conveyor 330, the solids contained in the slurry S are forced to the inner side of the bowl 320 and scraped out as a cake from the frustoconical part 322, as shown in the drawing, due to the rotation of the screw conveyor 330, and the cake to be dewatered is discharged from a cake discharge port 327A and a discharge chute 327B.

[0024] Meanwhile, clear liquid separated from the solids forms at one end of the bowl 320, and this liquid is discharged from a clear liquid discharge port 328A and a discharge chute 328B provided with a weir plate 325. The clear liquid from the discharge chute 328B has a high acetic acid concentration, is stored in a tank 8 via a conduit 8A, and is then returned to the oxidation reactor 1 by a pump 8B via a return channel 8C. Also, the clear liquid stored in the tank 8 is fed as necessary through a return channel 8D to a return channel 9D, to be described later, is guided to a group of dewatering and distillation columns 13, 13…, and is then separated into water vapor and acetic acid in the group of dewatering and distillation columns 13, 13… The acetic acid is returned to the oxidation reactor 1 via return channels 13B and 8C. The water vapor is condensed into wastewater by a condenser 14. The symbol 13A denotes a heater.

[0025] The cake discharged from the cake discharge chute 327B is charged into the first reslurry tank 6, is reslurried by a stirring device 6A with the aid of the filtrate of the first washing solution and a portion of the clear liquid supplied from the below-described first screen/bowl decanter centrifuge 400 to the first reslurry tank 6, and is then fed to the first screen/bowl decanter centrifuge 400 via a supply channel F2.

[0026] The same basic structure can be used as both the first and second screen/bowl decanter centrifuges referred to in the present invention, and this structure has a cylindrical screen unit with a screen on the wall surface of the smaller base of the frustoconical shape, which expands in relation to the decanter centrifuge. Specifically, to describe the structure using Fig. 4, in which the same symbols are used as in the first screen/bowl decanter centrifuge 400 in Fig. 1, the

casing 401a⁴ contains a bowl 420 with a cylindrical main body 421, a cylindrical screen unit 423 that has a screen 424 for filtering out solids on its wall and that is smaller than the cylindrical main body 421, and a frustoconical linking part 422 that links these two bodies; and a screw conveyor 430 having a spiraling screw blade 432 on the outer surface of a conveyor main body 431 in the bowl 420. The basic structure also includes rotation means for rotating the bowl 420 and the screw conveyor 430 at different speeds in the same direction.

[0027] In the rotation means, support axles 426A and 426B for the ends of the bowl 420, and support axles 433A and 433B for the ends of the corresponding screw conveyor 430 are coaxially supported on bearings 402A and 402B, rotational drive force is transmitted from a drive motor (not shown) to a pulley gear 403 to rotate the bowl 420, the rotational force is reduced with a reducing mechanism 404, and the screw conveyor 430 is rotated at a different speed than the bowl 420.

[0028] The slurry S supplied from the first reslurry tank 6 through the supply channel F2 is guided through a feed pipe 405 into a compartment 434 of the conveyor main body 431 in the first screen/bowl decanter centrifuge 400, and is then passed via a through-hole in the wall to be supplied between the cylindrical main body 421 and the screw conveyor 430.

[0029] Due to a relationship in which the slurry S is placed in the centrifugal force field in the bowl 420 and the screw conveyor 430, the solids contained in the slurry S are forced to the inner side of the bowl 420, are scraped out as a cake from the linking part 422, as shown in the drawing, due to the rotation of the screw conveyor 430, and are delivered to the cylindrical screen unit 423. In the cylindrical screen unit 423, the washing solution W pressurized by the centrifugal force and fed into the conveyor main body 431 through an external tube 472 disposed concentrically with the feed pipe 405 and provided with a washing solution supply tube 471 at one end thereof is distributed over the cake from washing solution sprayers 473, 473... disposed between the screw blades 432, whereby the cake is washed and filtered (centrifugally dewatered) therein due to the presence of the screen 424, and the filtrate of the first washing solution is discharged as a screen filtrate into a screen filtrate chute 429 as illustrated. The dewatering cake transferred to the screen unit 423 is discharged from a cake discharge port 427A and a discharge chute 427B disposed at the other end, while the clear liquid separated from the solids is

⁴ Translator's note: Indicated as "casing 401" in the original, which appears to be a typographical error.

discharged from a clear liquid discharge port 428A and a discharge chute 428B provided with a weir plate 425 and formed at the first end of the bowl 420.

[0030] The filtrate of the first washing solution recovered from the screen filtrate chute 429 is stored in a first washing filtrate tank 10 via a guiding tube 10A, and is then guided through a return channel 10C by a pump 10B to the first reslurry tank 6 as a reslurry medium.

[0031] Also, the clear liquid discharged from the clear liquid discharge chute 428B is stored in a first recovery tank 9 via a guiding tube 9A, a part thereof is then guided by a pump 9B through a return channel 9C to the first reslurry tank 6 as a reslurry medium, the residue is guided through a return channel 9D to a group of dewatering and distillation columns 13, 13… composed of a plurality of dewatering and distillation columns, the residue is separated into water vapor and acetic acid in the group of dewatering and distillation columns 13, 13…, the acetic acid is then returned to the oxidation reactor 1 via return channels 13B and 8C, and the water vapor is condensed into wastewater by a condenser 14.

[0032] The cake discharged from the cake discharge chute 427B is charged into the second reslurry tank 7. Part of the filtrate of the second washing solution and the clear liquid from the second screen/bowl decanter centrifuge 500 are fed to the second reslurry tank 7, are reslurried by a stirring device 7A, and are then fed to the second screen/bowl decanter centrifuge 500 via a supply channel F3.

[0033] In the second screen/bowl decanter centrifuge 500, solid-liquid separation and washing are performed in virtually the same manner as in the first screen/bowl decanter centrifuge 400, except that the second washing solution that is fed to washing solution sprayers 573, 573… is separately supplied pure water. Therefore, the operational configuration of this arrangement does not need to be described. The second washing solution may be the solution used as the washing solution in the purification/separation step.

[0034] The filtrate of the second washing solution recovered from the screen filtrate chute 529 is stored in a second washing filtrate tank 12 via a guiding tube 12A, is then guided by a pump 12B through a return channel 12C to the second reslurry tank 7, and is used as the reslurry medium.

[0035] The clear liquid recovered from a clear liquid discharge chute 528B is guided through a guiding tube 11A to a second recovery tank 11, part of the liquid is then guided by a pump 11B through a return channel 11D to a washing solution sprayer 473 as the first washing solution in the first screen/bowl decanter centrifuge 400, and the residue is fed to the second reslurry tank 7

via a return channel 11C. The rate at which the liquid is returned via these return channels can be appropriately controlled by providing a flow rate regulating valve or the like. When the second reslurry tank 7 is fed with a clear liquid from the second screen/bowl decanter centrifuge in addition to the supply of the filtrate of the second washing solution, it is known to result in extremely high washing efficiency.

[0036] The cake discharged from a cake discharge chute 527B of the second screen/bowl decanter centrifuge 500 is washed with pure water, the solvent is converted from an acetic acid base to a pure water base, and the material can therefore be supplied directly to the purification/separation step of the subsequent stage without passing through a dryer or silo. Therefore, equipment costs for a dryer and silo are reduced.

[0037] Also, the decanter centrifuge 300 and the first reslurry tank 6 in the above example can be omitted, and the acetic acid slurry from the crystallizer 2B can be fed directly to the first screen/bowl decanter centrifuge 400 via the supply channel F4, as shown in FIG. 5.

[0038] In the first screen/bowl decanter centrifuge 400 used in this embodiment, the filtrate of the first washing solution recovered from the screen filtrate chute 429 is stored in the first washing filtrate tank 10 via the guiding tube 10A, is then guided by the pump 10B through a return channel 10D to the group of dewatering and distillation columns 13, 13..., and is separated into water vapor and acetic acid in the group of dewatering and distillation columns 13, 13... The acetic acid is returned to the oxidation reactor 1 via the return channels 13B and 9D.

[0039] Meanwhile, the clear liquid from the clear liquid discharge chute 428B is stored in a first recovery tank 9 via the guiding tube 9A, is then returned by the pump 9B through the return channel 9D to the oxidation reactor 1, and, if necessary, is returned through a return channel 9E to the return channel 10D for guiding the filtrate of the first washing solution to the group of dewatering and distillation columns 13, 13...

[0040] However, when the filtrate of the first washing solution is returned to the oxidation reactor 1 via the group of dewatering and distillation columns 13, 13... in this manner, a regular screen/bowl decanter centrifuge has low crystal recovery efficiency because the crystals become admixed with the screen solution during screen filtration, and considerable leakage occurs. Therefore, the first washing solution must be fed to the supply side of the first screen/bowl decanter centrifuge 400, for example, to the supply channel F4, and the clear liquid from the clear liquid discharge chute 428B must be returned to the oxidation reaction system. In this case,

the filtrate of the first washing solution contains a large amount of water, so the clear liquid is diluted with water, a greater load is imposed on the group of dewatering and distillation columns 13, 13…, and it becomes impossible to recover or discharge the water in a smooth manner.

[0041] These drawbacks are substantially overcome in the embodiment shown in Fig. 1, wherein the decanter centrifuge 300 and the first reslurry tank are used. Since the filtrate of the first washing solution from the first screen/bowl decanter centrifuge 400 is returned to the first reslurry tank 6 and used as the reslurry medium, the leaked crystals are again fed to the first screen/bowl decanter centrifuge 400, and the crystal recovery efficiency increases. More specifically, in the present embodiment, the crystal recovery efficiency reaches about 99.95%, which is far superior to an extruding filter or other such type of continuous filtering device, which is about 70 to 90%. The clear liquid separated in the decanter centrifuge 300 is mostly acetic acid and contains a small amount of water as a byproduct of the oxidation reaction. The liquid can be directly returned to the oxidation reactor 1 by the pump 8B via the return channel 8C as previously described, and can also be smoothly returned to the oxidation reactor 1 with a small amount of water when directed to the group of dewatering and distillation columns 13, 13… Therefore, water can be recovered or discharged smoothly.

[0042] Instead of the above-mentioned decanter centrifuge, various continuous solid-liquid separators can be used as the solid-liquid separation means referred to in the present invention, which essentially should have a low rate of leakage of solids into the filtrate and should have satisfactory cake dewatering properties. For example, a BHS filter, a Young filter, an Oliver filter, or another such so-called rotary drum filter can be used.

[0043] Purification/Separation Step of the subsequent stage A CTA cake obtained by the crude aromatic dicarboxylic acid production/recovery step of the former stage as described above is fed to the purification/separation step wherein the crude aromatic dicarboxylic acid of the subsequent stage is subjected to a hydrogenation-reduction treatment to obtain high-purity aromatic dicarboxylic acid. Conventional methods, for example, a hydrogenation-reduction treatment, precision oxidation treatment, recrystallization treatment, or the like can be used in unaltered form as the purification/separation step of the subsequent stage. An example of hydrogenation-reduction treatment is described with reference to Fig. 2.

[0044] Specifically, a CTA cake obtained by converting a solvent from acetic acid to water is resuspended in the second reslurry tank ⁷⁵ and is then fed to a hydrogenation-reduction reactor 31. The impurities in the CTA are removed by a reduction treatment as a result of a hydrogenation reaction in the hydrogenation-reduction reactor 31. Specifically, 4CBA is converted to p-tolyllic acid or is partially converted to benzoic acid, and the tar-like colored substance is decolorized. Next, crystallization is performed by evaporation and cooling in an appropriate number (for example, five) of serially disposed crystallizers 32, the crystallized slurry is passed in sequence through a third rotary drum filter 33 that operates at increased pressure and a fourth drum filter 34 that operates at normal pressure, and the solid and liquid fractions are separated. A centrifuge may be used instead of the third rotary drum filter 33. In the solid-liquid separation means of the subsequent stage, the solid and liquid fractions are removed, the product is washed, the impurities are finally removed, and the material is dried in a dryer 35, kept in a silo 36, and stored as a finished product.

[0045] Preferred Screen/Bowl Decanter Centrifuge for the Present Invention On the other hand, the screen/bowl decanter centrifuge in the conventional example described above uses a washing solution sprayer and does not necessarily have high washing efficiency. The reason for this is described with reference to Fig. 11, wherein the same symbols are used as for the first screen/bowl decanter. The solids contained in the slurry supplied between the cylindrical main body 421 and the screw conveyor 430 via a through-hole in the wall of the compartment 434 are forced to the inner side of the bowl 420 in the centrifugal force field and are delivered in sequence to the cylindrical screen unit 423 by the screw blade 432, whereupon the solids are delivered as a cake C so as to be scraped together by the screw blade 432, and the surface level profile of the cake C slants at an incline towards the right side of the diagram as illustrated. After the cake C is delivered while the level profile is maintained, the inclination in the linking part 422 has an additional effect of further increasing the level incline, and the cake is scraped out and delivered to the screen unit 423 as the incline increases. Since a screen 424 is formed in the screen unit 423, filtration (centrifugal dewatering) is performed therein, and the screen filtrate is discharged as illustrated. Moreover, an uneven distribution of the cake C on the surface of the screen 424 creates an area on the forward delivery side in the section with the screw blades 432, 432 where there is no or virtually no cake C. When the washing solution has

⁵ Translator's note: The original reads "second reslurry tank 30," which is probably a typographical error.

been spread out in this state (including misting or spraying), the washing solution primarily flows along the incline of the surface of the cake C and cuts through the section of the screen 424 corresponding to the region with no cake C layer. The washing solution thus fails to pass through the cake C layer, and washing efficiency cannot be increased.

[0046] In view of this, the following embodiments (1) through (3), wherein means for making the cake C level uniform are provided to a conventional screen/bowl decanter centrifuge, are proposed for a screen/bowl decanter centrifuge with a high washing efficiency that is suitable for the manufacture of high-purity aromatic dicarboxylic acid according to the present invention.

[0047] (1) First Embodiment: Leveling and smoothing means whereby the pitch P3 of the screw blade 432A in the section extending from the linking part 422 to the first portion of the screen unit 423, or the pitch P2 of the screw blade 432A in the screen unit 423, is smaller than the pitch P1 of the screw blade 432 at one end (clear liquid expulsion side).

[0048] Fig. 6 shows the first embodiment of a preferred screen/bowl decanter centrifuge according to the present invention. Specifically, the pitch P2 of the screw blade 432A in the screen unit 423 is smaller than the pitch P1 of the screw blade 432 next to the linking part 422.

[0049] In conventional practice, the aforementioned pitches are the same. Moreover, in the first embodiment, the pitch of the screw blade 432A in the screen unit 423 is relatively small. The degree by which the pitch is made smaller is set primarily under conditions in which the cake C layer is formed entirely within the section of the screw blades 432A, 432A in the screen unit 423, and is made as level as possible (for example, about 95 to 35%).

[0050] An option may also be considered in which it is better for the pitches of all the screw blades to be smaller, in which case an optimal screw blade pitch still exists in terms of the treated amount, solid-liquid separability, and other properties of the apparatus of a given size, so the screw blade 432 must be provided at this pitch to the cylindrical main body 421 and the linking part 422. By contrast, such factors are few in the case of the screen unit 423, and it is possible to vary the pitch of the screw blade 432A.

[0051] The first embodiment does not exclude aspects in which the pitches of the screw blades 432, 432 in the cylindrical main body 421 and the linking part 422 differ from each other, and the first embodiment is limited only in terms of the relationship between the pitch of the screw blade 432A in the screen unit 423 and the pitch of the screw blade 432 next to the linking part 422.

[0052] Also, as long as the pitch of the screw blade 432A in the screen unit 423 is smaller than the pitch of the screw blade 432 next to the linking part 422, it is possible to reduce the pitch in at least one section rather than decreasing the pitches between all the screw blades 432A, 432A, and 432A, 432A... in the screen unit 423. Furthermore, the pitch of the screw blades 432A in the screen unit 423 can be varied, and can, for example, be sequentially reduced towards the other end (the side with the cake discharge port 427A).

[0053] Decreasing the pitch P3 in the section (including the screen unit) extending from the linking part 422 to the first portion of the screen unit 423 (not shown) is also included in the first embodiment.

[0054] Thus, in the first embodiment as well as in the following second and third embodiments, the cake washing efficiency can be improved because the washing solution from the washing solution sprayers 473, ...473 invariably passes though the entire cake C without creating a section in which the surface of the screen 424 is exposed and is devoid of a layer of the cake C between the screw blades 432, as in conventional practice.

[0055] (2) Second Embodiment: Leveling and smoothing means wherein a screen may or may not be provided between the screw blade 432A next to the linking part 422 in the screen unit 423 and the adjacent screw blade 432A, but the screen 424 is blinded and a washing solution sprayer is provided facing the cake C surface.

[0056] Fig. 7 shows the second embodiment, wherein a blind member B is disposed between the screw blade 432A next to the linking part 422 in the screen unit 423 and the adjacent screw blade 432A.

[0057] In this case, the section impermeable to the washing solution in the screen unit 423 can have a pitch of 1/5 to 2 times the pitch P2 of the screw blade 432A, or of about 20 to 600 mm. In the example in Fig. 7, an area about 1.5 times the pitch serves as the section impermeable to the washing solution.

[0058] In the blind arrangement, it is possible to cover the screen 423 with a separate member while the screen is being formed, rather than directly extend the structural members of the linking part 422 by the corresponding length of the screen 424.

[0059] In the second embodiment, the region with the blind member B to which the washing solution is fed is not subjected to dewatering. Therefore, the amount of fluid in the cake C increases in this portion, and the cake C expands into the entire space between the screw

blades 432A, 432A as a result of the increased fluidity. The smoothed level profile of the cake C is maintained even as the cake moves to the other end (the side with the cake discharge port 427A), and the cake washing efficiency is therefore improved in comparison with conventional practice.

[0060] (3) Third Embodiment: Leveling and smoothing means wherein the solid surface level between the screw blade 432A and the adjacent screw blade 432A is controlled and smoothing members M are provided to the screw conveyor 430.

[0061] Figs. 8 and 9 show the third embodiment. Specifically, the solid surface level between the screw blade 432A and the adjacent screw blade 432A is kept smooth, and smoothing members M are provided to the screw conveyor 430.

[0062] The smoothing members M may be fixed to the conveyor main body 431, or they may be fixed to the screw blade 432A. In either case, the distal edges of the smoothing members M in the radial direction are fixed to leave a specific gap with the inner surface of the screen unit 423. Instead of being parallel with the axial direction of the apparatus, as shown in Fig. 8, the distal edges of the smoothing members M in the radial direction may form a straight or curved line at an appropriate angle. The smoothing members M may extend in the axial direction the entire length of the section with the screw blades 432A, 432A, or only part of the length, as shown in Fig. 8.

[0063] According to the third embodiment, the smoothing members M rotate along with the rotation of the screw conveyor 430, and the cake C on the screen unit 423 is smoothed into an entirely uniform thickness between the screw blades 432A, 432A.

[0064] (Other) In the second embodiment, it is essential that the washing solution be spread out, whereas in the other embodiments spreading out of the washing solution is desirable but not essential. Specifically, this is because smoothing the level of the cake C has the effect of facilitating centrifugal dewatering compared with when the level is not smoothed (the thickness of the layer when not smoothed offers resistance and reduces the centrifugal dewatering potential). In either case, in the method and apparatus for manufacturing high-purity terephthalic acid according to the present invention, the cake must be washed by the washing solution, and the first through third embodiments described above can therefore be suitably used.

[0065] A working example of the method for manufacturing high-purity terephthalic acid according to the present invention is shown below, and the effects of the present invention are described.

[0066] <Working Example> Fig. 10 shows an operating example of the CTA production/separation step according to the present invention. The weight ratio of the acetic acid, indicated as "A.A." in the diagram, in the slurry supplied from the oxidation reaction system to the decanter centrifuge 300 can be such that the ratio, which is initially 58.60%, is then changed to A.A. = 14.82% for the slurry in the first reslurry tank 6, to A.A. = 1.38% for the slurry in the second reslurry tank 7, and to A.A. = 0.09% for the cake discharged from the second screen/bowl decanter centrifuge 500. It should be noted that A.A. contains many impurities due to the reaction.

[0067] Therefore, it is possible to dispense with the need for the conventionally used step of drying the material in a dryer and storing it in a silo, and to continuously perform the CTA manufacturing/recovery step and the purification/separation step.

[0068] Also, the above descriptions were focused on the manufacture of terephthalic acid, but those skilled in the art should easily understand without the aid of any working examples that the present invention can also be used in the manufacture of isophthalic acid, naphthalene dicarboxylic acid, and other such aromatic dicarboxylic acids.

[0069]

[Effect of the Invention] As described above, the present invention is advantageous in making it possible to dispense with the need for the drying step with a CTA dryer and the storage step with a CTA silo that have been used in conventional practice, to reduce the necessary equipment costs, to increase manufacturing efficiency by combining the preceding and subsequent stages into a single continuous step, and to improve the purity of the resulting crystals.

[Procedural Amendment 2]

[Document Amended] Specification

[Section Amended] Brief Description of the Drawings

[Amendment Method] Change

[Details of Amendment]

[Brief Description of the Drawings]

[Figure 1] A flowsheet of the terephthalic acid production/recovery step according to the present invention

[Figure 2] A flowsheet of the purification/separation step of the subsequent stage

[Figure 3] A longitudinal cross-sectional view showing an outline of the decanter centrifuge

[Figure 4] A longitudinal cross-sectional view showing an outline of the first screen/bowl decanter centrifuge

[Figure 5] A flowsheet showing another embodiment of the terephthalic acid production/recovery method according to the present invention

[Figure 6] A schematic enlarged longitudinal sectional view showing a first embodiment of a screen/bowl decanter centrifuge suitable for the present invention

[Figure 7] A schematic enlarged longitudinal sectional view showing a second embodiment of a screen/bowl decanter centrifuge suitable for the present invention

[Figure 8] A schematic enlarged longitudinal sectional view showing a third embodiment of a screen/bowl decanter centrifuge suitable for the present invention

[Figure 9] A cross-sectional side view thereof

[Figure 10] An explanatory diagram of an operating example

[Figure 11] A schematic enlarged longitudinal sectional view of Fig. 4

[Figure 12] A schematic flowsheet of a conventional example

[Key]

1: oxidation reactor, 2A, 2B: evaporation crystallizer, 6: first reslurry tank, 7: second reslurry tank, 9: first recovery tank, 10: first washing filtrate tank, 11: second recovery tank, 12: second washing filtrate tank, 13: dewatering and distillation columns, 300: decanter centrifuge, 400: first screen/bowl decanter centrifuge, 500: second screen/bowl decanter centrifuge